

JAPANESE
INDUSTRIAL
STANDARD

Translated and Published by
Japanese Standards Association

JIS K 0090 : 1998

Method for determination of
phosgene in flue gas

ICS 13.040.40; 71.040.40

Descriptors : carbonyl chloride, exhaust gases, gas analysis, industrial air
pollutants, industrial wastes, sampling methods, spectrophotometry,
gas chromatography, determination of content

Reference number : JIS K 0090 : 1998 (E)

K 0090:1998

Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of International Trade and Industry through deliberations at Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law:

Date of Establishment: 1975-08-01

Date of Revision: 1998-03-20

Date of Public Notice in Official Gazette: 1998-03-20

Investigated by: Japanese Industrial Standards Committee

Divisional Council on Environment

Technical Committee on Air Quality

JIS K 0090:1998, First English edition published in 1998-10

Translated and published by: Japanese Standards Association
4-1-24, Akasaka, Minato-ku, Tokyo, 107-8440 JAPAN

In the event of any doubts arising as to the contents,
the original JIS is to be the final authority.

© JSA 1998

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

Printed in Japan

Method for determination of phosgene in flue gas

1 Scope This Japanese Industrial Standard specifies the method for determination of phosgene (carbonyl chloride) in flue gas.

Remarks 1 In this Standard, flue gas means ones which is generated by being accompanied with manufacturing processes of various chemical products, combustion, other chemical reactions and manufacturing process using phosgene, etc., and exhausted to flue, chimney or duct.

2 The normative references are shown in Attached Table 1.

2 Common items The common items of chemical analysis, sampling methods of flue gas and molecular absorptiometric analysis shall be in accordance with **JIS K 0050**, **JIS K 0095** and **JIS K 0115**, respectively.

3 Classification of analytical methods and their outlines Classification of analytical methods and their outlines shall be as shown in Table 1.

Table 1 Classification of analytical methods and their outlines

Classification of analytical method	Outlines of analytical method			Applicable condition
	Summary	Sampling	Range of determination vol ppm	
Diphenylurea ultraviolet absorptiometry	After absorbing phosgene in the sample gas into absorbent, generated diphenylurea is extracted by solvent and the absorbance (257 nm in wavelength) of this extracted solution is measured.	Absorption bottle method Absorbent : Aniline solution (2.5 g/l, pH 7 to pH 9) Absorption volume : 10 ml × 1	0.1 to 5 (20 l)	In accordance with 5.1

4 Sampling method of sample gas Sampling position of gas for analysis shall be selected the point where the typical gas can be collected and, after collected at least twice in the same position within adjacent time, and these gases shall be analyzed respectively.

4.1 Sampling method Absorption bottle method

4.2 Reagents and preparation method of reagent solution

(1) **Reagents**

- (a) **Water** A2 grade specified in **JIS K 0557**.
- (b) **Hydrochloric acid** Specified in **JIS K 8180**.
- (c) **Sodium hydroxide** Specified in **JIS K 8576**.
- (d) **Aniline** Specified in **JIS K 8042**.

- (2) **Preparation method of reagent solution (absorbent)** Dissolve 25 g of aniline in hydrochloric acid (1 mol/l) to dilute to 1 l. Take 100 ml of this solution at the time of use and add sodium hydroxide (1 mol/l) to adjust pH 7 to pH 9 and dilute it to 1 l.

4.3 Apparatus and device

- (1) **Absorption bottle** Only one absorption bottle which has 40 ml to 50 ml in volume and is shown in Fig. 1 as an example shall be used.

Remarks: This absorption bottle shall be as the same as that of Fig. 3 (d) in JIS K 0095 or equivalent to the example. Though the absorption bottle may be attached with nozzle [Fig. 3 (b)], inside diameter of nozzle in this case shall be 1 mm and connected 2 nozzles in series.

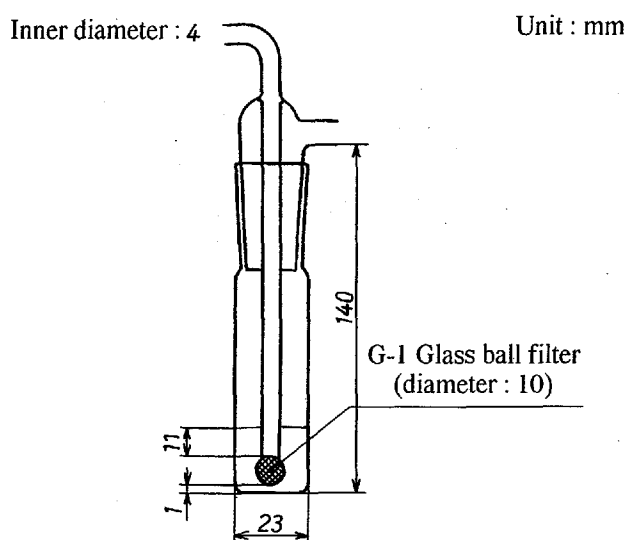
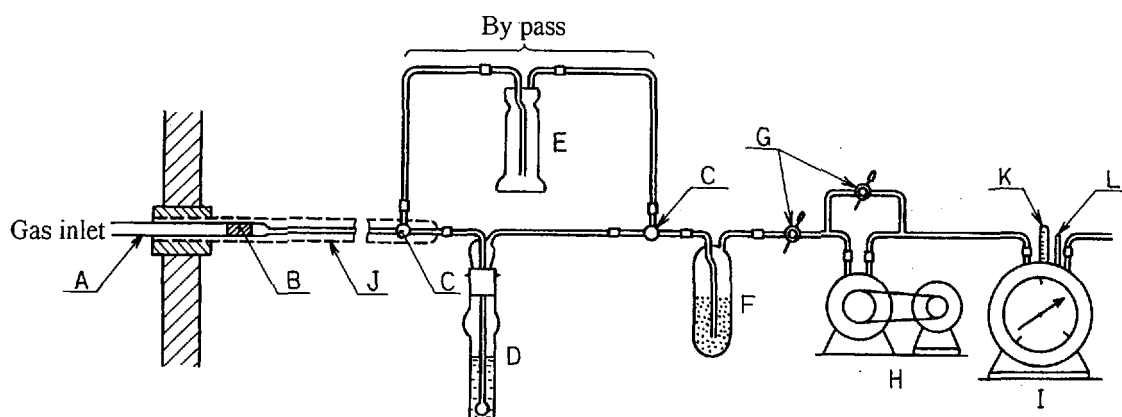


Fig. 1 An example of absorption bottle

- (2) **Gas sampling apparatus** The apparatus shall be constructed as shown in Fig. 2 for example and provided with the following conditions. This apparatus is the construction of Fig. 2 (a) in JIS K 0095 [absorption bottle method (in case of measuring the volume of sample gas by gas meter)].

- (a) Gas sampling apparatus shall be made of such materials that are not corroded by co-existing components in flue gas. For example, glass, stainless steel, quartz glass and fluoride resin shall be used.
- (b) In order to prevent against mixing dust into sample gas, filter medium without alkali shall be inserted into a suitable place of gas sampling apparatus ⁽¹⁾.

Note ⁽¹⁾ As phosgene has tendency to decompose easily at high temperature, heating shall be avoided stubbornly and, in an unavoidable case such as much condensation of water, the heating temperature shall be 100 °C or lower.



- A : Gas sampling tube F : Trap (filling up glass wool) J : Heater
 B : Filter medium G : Flow rate adjusting cock K : Thermometer
 C : Three way stopcock H : Closed type suction pump (1 to 5 l/min) L : Pressure gauge
 D : Absorption bottle I : Wet type gas meter (1 to 5 l/per revolution)
 E : Washing bottle [put in 50 ml of sodium hydroxide solution (200 g/l)]

Fig. 2 An example of gas sampling apparatus

4.4 Sampling gas The method of sampling gas shall be as follows.

- (1) Put 10 ml of absorbent into absorption bottle (²).

Note (²) In case of absorption bottle having larger inside diameter than that of Fig. 1, the distance of absorbent level in still from glass wool filter shall be more than 3 cm. In case of the distance smaller than 3 cm, add absorbent to maintain this level.

In case of using absorption bottle with nozzle, the distance of nozzle from absorbent level in still condition shall be more than 6 cm. In case of the distance lower than 6 cm, add absorbent to maintain this level.

- (2) Place the gas sampling apparatus at a position as near the sampling position as possible. Prior to introducing gas into absorption bottle, substitute sufficiently with sample gas in the piping.
- (3) Keep the flow rate of sample gas not exceeding 0.7 l/min, and take 20 l of the sampling volume, and increase or decrease this volume according to the concentration of phosgene in sample gas. At the same time as the measurement of sample gas volume, measure the temperature and pressure of gas in flow meter.

5 Determination of phosgene

5.1 Applicable requirements In case of coexisting with acidic gas such as chlorine and nitrogen dioxide, as the results of analysis are affected positively, this method shall be applied when these effects can be ignored or eliminated.